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IS 2600-2 (1988): Methods of chemical analysis of zinc and zinc base alloys for die castings, Part 2: Determination of copper, iron, lead and cadmium by atomic absorption spectrophotometric method [MTD 9: Lead, Zinc, Cadmium, Tin, Antimony and their Alloys]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**METHODS OF
CHEMICAL ANALYSIS OF ZINC AND
ZINC BASE ALLOYS FOR DIE CASTINGS**

**PART 2 DETERMINATION OF COPPER, IRON, LEAD AND CADMIUM BY
ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD**

(First Revision)

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ZINC AND ZINC BASE ALLOYS FOR DIE CASTINGS

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(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 25 July 1988, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 2600 was published in 1964 and has been revised (into parts) on the basis of implementation of the standard by the manufacturers and testing laboratories. Part 2 covers the atomic absorption spectrophotometric methods for the various constituents whereas in Part 1 the spectrophotometric methods have been

prescribed with the publication of these parts, the method of analysis for relevant constituents published in earlier version will be superseded. The analysis of constituents not covered in the above parts will be covered in subsequent parts.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard (Part 2) covers the method for determination of copper, iron, lead and cadmium by atomic absorption spectrophotometric method (AAS) in the range as specified in IS : 713-1981* and IS : 742-1981†.

2. SAMPLING

2.1 Sample shall be drawn and prepared in accordance with the procedure laid down in IS : 1817-1961‡.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified, analytical grade reagents and double distilled water shall be employed in the test.

4. DETERMINATION OF COPPER, IRON, LEAD AND CADMIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRIC (AAS) METHOD

4.1 The sample is dissolved in hydrochloric nitric acid mixture and suitable aliquot is

aspirated for atomic absorption using air-acetylene flame. Copper, cadmium, lead and iron are determined by standard addition method.

4.2 Reagents

4.2.1 Hydrochloric Acid — rd = 1.16 (conforming to IS : 265-1976*).

4.2.2 Dilute Nitric Acid — 40 percent (v/v).

4.2.3 Standard Solution of Copper (100 ppm) — Transfer 1.00 g of copper (purity 99.99 percent) to a 250-ml beaker and add 15 ml of dilute nitric acid (1 : 3), warm gently until copper is dissolved. Cool, add 20 ml water and boil again. Transfer the solution to 1 litre volumetric flask, and make up and mix. Take 10 ml of the solution and dilute to 100 ml (see Note).

4.2.4 Standard Solution of Iron (100 ppm) — Transfer 1.00 g of iron (purity 99.99 percent) to a 100-ml beaker. Add 10 ml of dilute hydrochloric acid (1 : 1) and 1 ml of bromine water. Boil until excess bromine is expelled. Add 20 ml of concentrated hydrochloric acid and transfer to 1 litre volumetric flask. Cool and make up to the volume. Take 10 ml of the solution and dilute to 100 ml in a volumetric flask (see Note).

*Specification for zinc base alloy ingots for die castings (second revision).

†Specification for zinc base alloys die castings (second revision).

‡Methods of sampling non-ferrous metals for chemical analysis.

*Specification for hydrochloric acid.

4.2.5 Standard Solution of Lead (100 ppm) — Transfer 1.00 g of lead (purity 99.99 percent) to a 250-ml beaker, and add 50 ml of dilute nitric acid (1 : 1) and cover. Boil to remove oxides of nitrogen. Cool and transfer 1 litre volumetric flask. Add 50 ml concentrated nitric acid and cool. Make up the volume and transfer to a polyethylene bottle. Take 10 ml of the solution and dilute to 100 ml in a volumetric flask (see Note).

4.2.6 Standard Solution of Cadmium (100 ppm) — Transfer 1.00 g of cadmium (purity 99.99 percent) to a 250-ml beaker, and add 25-ml dilute nitric acid (1 : 1) and 5-ml concentrated hydrochloric acid. Boil to remove oxides of nitrogen. Cool and transfer to 1 litre volumetric flask. Add 50 ml concentrated hydrochloric acid and make up. Take 10 ml of the solution and dilute to 100 ml in a volumetric flask (see Note).

NOTE — Dilution should be made as and when required.

4.3 Procedure

4.3.1 Weigh 10.00 g sample in a 500-ml conical flask. Add 30-ml concentrated hydrochloric acid and 20-ml dilute nitric acid. Cover with watch glass. When the reaction ceases, boil to expel nitrous fumes. Cool and (filter if any suspended particles are observed) transfer the solution to 100-ml volumetric flask and make up.

4.3.2 Take 10 ml of aliquot into two separate 100-ml volumetric flasks (flasks B and C). Make up the volume of flask B. To flask C, add 1 ml (see Note) each of standard solution of copper, iron, cadmium and lead, and make up.

NOTE — Suitable standard addition should be made so that the difference in absorbance for the solutions in C and B flasks fall between 0.1 and 0.5.

4.3.3 Blank — Use the same quantity of reagents mentioned in 4.3.1 and make up. Take 10-ml aliquot of the solution and transfer to 100-ml volumetric flask and make up. Mark this solution as flask A.

4.3.4 Determination of Copper — Align copper hollow cathode lamp. Feed lamp current, set slit width as recommended in the manual of the model of AAS. Set wavelength 324.75 nm, optimize with fine adjustment for maximum transmission. Put on air-acetylene flame. Aspirate double distilled water and make the final settings of burner, lamp and wavelength for zero absorbance. Aspirate blank solution (A) and record absorbance, if any. Normally absorbance remains zero. Aspirate the sample (B) and record absorbance average of six readings. Then aspirate sample and standard solution (C), and record absorbance average of six readings.

4.3.4.1 Calculation

$$\text{Copper, percent} = \frac{(b - a)}{(c - b) \times 100}$$

where a , b and c are the absorbances of solutions A, B and C respectively.

4.3.5 Determination of Cadmium — Align cadmium hollow cathode lamp. Set slit width and feed lamp current mA as recommended in the operational manual of the model of AAS. Set wave length 228.8 nm and optimize with fine adjustments for maximum transmission. Put on air-acetylene flame. Aspirate double distilled water and make the final settings for maximum transmittance and zero absorbance. Aspirate blank solution (A) and record absorbance, if any. Aspirate the sample solution (B) and record absorbance, an average of six readings. Finally aspirate sample and standard solution (C) and record absorbance reading an average of six readings.

4.3.5.1 Calculation

$$\text{Cadmium, percent} = \frac{(b - a)}{(c - b) \times 100}$$

where a , b and c are the absorbances of solutions A, B and C respectively.

4.3.6 Determination of Lead — Align the hollow cathode lamp of lead in optical path. Set recommended slit width and feed lamp current mA as recommended in operational manual of the AAS model. Set wavelength 217 nm and optimize with fine adjustments for maximum transmittance. Put on air acetylene flame, make the final settings for maximum transmission and zero absorbance. Aspirate blank solution (A) and record absorbance. Then aspirate the sample (B) recording an average of six absorbance recordings. Finally aspirate sample and standard solution (C), and record average of six absorbance readings.

4.3.6.1 Calculation

$$\text{Lead, percent} = \frac{(b - a)}{(c - b) \times 100}$$

where a , b and c are the absorbances of solutions A, B and C respectively.

4.3.7 Determination of Iron — Align hollow cathode of iron in the optical path. Set recommended slit width and feed lamp current as recommended in the operational manual of AAS. Set wavelength 248.33 nm. Put on air-acetylene flame and aspirate double distilled water, make the final setting for maximum transmission and zero absorbance. Aspirate blank solution (A) and record absorbance, if any.

Aspirate the sample solution (B) and record absorbance, an average of six readings. Finally aspirate sample and standard solution (C), and record an average of six absorbance readings.

4.3.7.1 Calculation

$$\text{Iron, percent} = \frac{(b - a)}{(c - b) \times 100}$$

where a , b and c are the absorbances of solutions A, B and C respectively.